

(12) UK Patent Application (19) GB (11) 2 280 111 (13) A

(43) Date of A Publication 25.01.1995

(21) Application No 9414227.0

(22) Date of Filing 14.07.1994

(30) Priority Data

(31) 08093118

(32) 16.07.1993

(33) US

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(51) INT CL<sup>6</sup>

A61K 7/32

(52) UK CL (Edition N )

A5B BFG

(56) Documents Cited

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(58) Field of Search

UK CL (Edition M ) A5B BFG

INT CL<sup>6</sup> A61K 7/32 7/34 7/36 7/38

ONLINE DATABASES: WPI, CLAIMS

(54) Antiperspirant

(57) Clear gel antiperspirant compositions comprise an antiperspirant active, a dihydric alcohol as primary solvent, a cosolvent of polyethylene glycol, water, and/or glycerine, a buffering agent, and a gelling agent. The antiperspirant active is e.g. an aluminium salt or an aluminium and/or zirconium complex.

Typical dihydric alcohol components are di or propylene glycol, hexylene glycol and/or pentanediol.

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ANTIPERSPIRANT COMPOSITION  
AND METHOD OF USE THEREOF

5                    Field of the Invention

                  This invention relates generally to antiperspirant  
compositions and, in particular, to a stable, clear  
antiperspirant composition that is available in stick  
10 form.

Background of the Invention

                  Antiperspirant stick compositions represent an  
important and growing segment of the antiperspirant and  
15 deodorant market as the demand for environmentally  
unfriendly product forms such as aerosols wanes. Prior  
art antiperspirant stick compositions often leave an  
unsightly and unpleasant white residue on the skin.  
This residue film can leave a mark or stain on clothing  
20 worn in the area where the composition has been applied.

                  In recent years there has been a trend towards  
clear products in many fields as clear products are  
perceived to be less damaging to the environment. While  
clear deodorant stick compositions have been formulated  
25 successfully, the formulation of a clear antiperspirant  
gel stick composition has proved to be more problematic.

                  The temperatures required for processing gel stick  
compositions are high. Furthermore, typical  
antiperspirant active agents are aggressive acids with a  
30 pH of less than 5. In general, gellants degrade at high  
temperatures and at a pH of less than about 7.

                  It would, therefore, be desirable to provide a  
stable, clear antiperspirant gel stick composition.

Summary of the Invention

Generally speaking, a stable, clear antiperspirant composition having an antiperspirant active, a dihydric  
5 alcohol as a primary solvent, a cosolvent such as low molecular weight polyethylene glycol (l.m.wt. PEG), water, and/or glycerine, a buffering agent, and a gelling agent is provided. The antiperspirant active is preferably an aluminium salt or an aluminium and/or  
10 zirconium complex. A method for reducing perspiration using the composition is also provided.

The invention accordingly comprises a composition of matter possessing the characteristics, properties, and the relation of components which will be exemplified  
15 in the composition hereinafter described, and the scope of the invention will be indicated in the claims.

Detailed Description of the Preferred Embodiments

20 According to the present invention there is provided a stable, clear gel antiperspirant composition having no volatile organic compounds, an acceptable odour, and a satisfactory clarity comprising:  
an antiperspirant active;  
25 a dihydric alcohol solvent;  
a cosolvent selected from low molecular weight polyethylene glycol, water, glycerine and mixtures thereof;  
a buffering agent; and  
30 a gelling agent.

In the absence of fragrancng agents, the composition of the invention has an acceptable odour. The odour of a gel composition is a measure of stability  
35 since an almond odour that is characteristic of

benzaldehyde will be present if there is degradation of the gelling agent. In general, an odour rated at less than or equal to 3 on a scale of 0 to 5 wherein 0 represents no odour and 5 represents a distinctive odour is considered acceptable.

The composition also has a satisfactory clarity. Clarity is rated on a scale of 0 to 5 wherein 0 represents complete clarity and 5 represents opacity. A clarity of less than or equal to 3 is considered satisfactory. In addition, it is preferable if the composition does not salt, i.e. become gritty, when it is stored for a period of time.

Effective antiperspirant active agents are aggressive acids. The antiperspirant active is generally an aluminium astringent antiperspirant salt or an aluminium and/or zirconium astringent complex having a pH of less than or equal to about 5. Aluminium salts are not effective antiperspirant active agents at a pH of greater than about 5 because the salt breaks down into insoluble components. This breakdown of the salt into insoluble components also causes the composition to become cloudy, which is especially undesirable with respect to the formulation of a clear composition.

The inherent pH of an unbuffered antiperspirant active is between about 3.5 and 4.4. In the composition provided in accordance with the invention, which is buffered, the pH of the antiperspirant active is preferably between about 4 and 4.9 and more preferably, between about 4.5 and 4.8.

The antiperspirant active is preferably selected from aluminium halides, aluminium hydroxyhalides, aluminium sulfates, zirconyl oxyhalides, zirconyl hydroxyhalides, and mixtures thereof. In a more preferred embodiment, the antiperspirant active is aluminium chlorohydrate, which is a solid complex of

aluminium chlorohydrate and propylene glycol. It is available from Reheis Chem. Co. of Berkeley Heights, NJ and is sold under the trade name REHYDROL®.

5 The antiperspirant active is present in an amount sufficient to prevent perspiration and is generally present in an amount between about 10% and 30% by weight of the composition. In a preferred embodiment, the antiperspirant active is present in an amount between about 10% and 25% by weight of the composition; in a  
10 more preferred embodiment, the antiperspirant active is present in an amount between about 15% and 25% by weight; and, in a most preferred embodiment, the antiperspirant active is present in an amount between about 18% and 25% by weight.

15 A significant percentage by weight of the composition is a primary solvent for the antiperspirant active agent. The primary solvent is a dihydric alcohol preferably selected from propylene glycol, hexylene glycol, dipropylene glycol, pentanediol, and mixtures  
20 thereof. In a more preferred embodiment, the primary solvent is propylene glycol.

The primary solvent is present in an amount sufficient to dissolve both the antiperspirant active agent and the gelling agent and is generally present in  
25 an amount between about 10% and 70% by weight of the composition. In a preferred embodiment, the solvent is present in an amount between about 15% and 70% by weight.

A cosolvent for the gelling agent is also  
30 present. The cosolvent is selected from low molecular weight polyethylene glycol, water, glycerine, and mixtures thereof. The cosolvent is typically present in an amount between about 1% and 30% by weight of the composition.

When low molecular weight polyethylene glycol (l.m.wt. PEG) is used in the composition, it is effective to inhibit salting since materials that would cause the composition to salt or become gritty are generally soluble in l.m.wt. PEG. The l.m.wt. PEG also functions as an emollient to cause the gel composition to have a gliding effect on skin. Additionally, the PEG causes the composition to feel "less sticky" than it would if no l.m.wt. PEG were present.

5 The l.m.wt. PEG used in the composition as an anti-salting agent is a liquid at room temperature with an average molecular weight of less than or equal to about 1,000. In a preferred embodiment, the l.m.wt. PEG has an average molecular weight of less than or equal to  
10 about 600 and in an especially preferred embodiment, the l.m.wt. PEG has an average molecular weight of less than or equal to about 400.

The l.m.wt. PEG is generally present in the composition in an amount between about 1% and 20% by weight of the composition. In a preferred embodiment,  
15 the l.m.wt. PEG is present in an amount between about 5% and 15% by weight of the composition and in an especially preferred embodiment, the l.m.wt. PEG is present in an amount between about 7% and 13% by weight.

20 Water can be included in the composition as an alternate or additional cosolvent. Water functions to prevent syneresis or sweating of the composition on exposure to air. In addition, water can also be used to give the composition a dry feel as opposed to a sticky  
25 feel. When water is present in the composition, there must be sufficient gelling agent to cause the composition to solidify at a temperature of less than or equal to about 90°C so that the water does not boil off during processing.

The amount of water used should be between about 1% and 20% by weight of the composition. In a preferred embodiment, the amount of water is between about 3% and 15% by weight of the composition; in a more preferred embodiment, the amount of water is between about 5% and 15% by weight; and, in an even more preferred embodiment, the amount of water is between about 5% and 13% by weight.

A third alternate or additional cosolvent is glycerine. When used, glycerine functions as an emollient to give the composition a smooth, gliding feel on the skin.

The amount of glycerine used should be between about 1% and 20% by weight of the composition. In a preferred embodiment, the amount of glycerine is between about 3% and 15% by weight of the composition and in an especially preferred embodiment, the amount of glycerine is between about 5% and 13% by weight.

A buffering agent or system is used in the composition of the invention to prevent wide pH swings. Ideally, the buffering agent is an acid/base system having a  $pK_a$  of from about 3.5 to about 5, preferably from about 4.5 to about 5.

The buffering agent is selected from phosphate buffers such as sodium or potassium phosphate, phosphoric acid, potassium hydroxide, potassium acid phthalate, triethanolamine, citric acid, sodium citrate, aluminium lactate, lactic acid and mixtures thereof. In a preferred embodiment, a buffer solution of citric acid, sodium citrate and potassium hydroxide is used.

In general, an inorganic buffering agent is more heat stable. However, phosphate buffers, for example, increase the amount of salting or grittiness that occurs on standing due to breakdown of the antiperspirant active salt into insoluble components.

The greater the amount of buffering agent in the composition, the better the stability of the composition and the better the pH is controlled. On the other hand, lower amounts of buffering agents are preferred in order  
5 to increase the clarity of the solution and decrease the formation of insoluble components.

The buffering agent is generally present in an amount between about 0.1% and 3% by weight of the composition of the invention. In a preferred  
10 embodiment, the buffering agent is present in an amount between about 0.2% and 2% by weight of the composition and in an especially preferred embodiment, the buffering agent is present in an amount between about 0.2% and 1% by weight.

15 A material that functions as a gelling agent at a low pH and at high temperatures of up to about 140°C is also present in the composition. The gelling agent is preferably selected from dibenzylidene sorbitol resin, derivatives of dibenzylidene sorbitol resin, and  
20 mixtures thereof. In a preferred embodiment, the gelling agent is dibenzylidene sorbitol resin.

In general, dibenzylidene sorbitol resin and its derivatives degrade or decompose under acidic conditions, specifically at a pH of less than about 7,  
25 and at high temperatures to form sorbitol and benzaldehyde. This decomposition can be confirmed by the presence of benzaldehyde, which has a characteristic sweet odour that is reminiscent of freshly crushed bitter almonds. However, the presence of the buffering  
30 agent in the composition of the invention slows the degradation of the dibenzylidene sorbitol resin considerably, thereby permitting the composition to have little or no almond odour, which is considered acceptable.

The gelling agent is preferably present in an amount between about 1% and 5% by weight of the composition. In a preferred embodiment, the gelling agent is present in an amount between about 2% and 4% of the composition.

Other optional components such as thickening agents, liquid emollients, emulsifiers, dyes and fragrancings agents can also be included in the composition in amounts of between about 0.1% and 10% by weight of the composition, more preferably, in amounts between about 0.1% and 5% by weight.

Optional thickening agents include, for example, solid esters. Solid esters include, but are not limited to, high molecular weight polyethylene glycol, polyethylene glycol laurates, polyethylene glycol cocoates, polyethylene glycol myristates and solid polyethylene glycol stearates.

One exemplary thickening agent is high molecular weight polyethylene glycol (h.m.wt. PEG). As used herein, h.m.wt. PEG is polyethylene glycol with an average molecular weight of over about 1,000. High molecular weight polyethylene glycol is a solid at room temperature and therefore, is not useful as an antiscalting agent. The amount of h.m.wt. PEG needed to perform a thickening agent function decreases as the average molecular weight of the h.m.wt. PEG increases.

When h.m.wt. PEG is used in the composition, it is incorporated in an amount between about 0.5% and 8% by weight. In a preferred embodiment, the h.m.wt. PEG is used in an amount between about 0.5% and 5% by weight of the composition and, in a more preferred embodiment, the h.m.wt. PEG is used in an amount between about 0.5% and 2% by weight.

When used, the amount of h.m.wt. PEG determines the amount of buffering agent that is required. For

example, the optimum amount of buffering agent is in inverse proportion to the amount of high molecular weight polyethylene glycol in the composition. When a large amount of high molecular weight polyethylene glycol is used, a relatively small amount of buffering agent is required; conversely, when a small amount of h.m.wt. polyethylene glycol is used, a relatively large amount of buffering agent is required.

The clear antiperspirant gel compositions of the invention are prepared by dissolving the antiperspirant active resin agent in a first portion of the dihydric alcohol solvent, e.g. propylene glycol, or in a mixture of low molecular weight PEG and a portion of the dihydric alcohol solvent to yield a first solution.

A second portion of the dihydric alcohol solvent, the cosolvent, and at least part of the buffering agent are dissolved separately and heated to a temperature between about 95° and 140°C, preferably between about 110° and 130°C, to yield a second solution. The gelling agent is added to the second solution.

The first solution is heated to a maximum temperature of 100°C and added to the second solution so that the temperature does not drop below about 95°C. If water is used as the cosolvent, it can be heated to a temperature of up to about 95°C and added at this point rather than earlier as indicated above. Any other optional ingredients can also be added at an elevated temperature. The solution is packed at about 90°C and allowed to cool.

The compositions prepared in accordance with the invention are evaluated for odour, clarity and salting. The following scale was used to evaluate odour:

0	No odour
1	Threshold odour
2	Very slight odour

- 3 Slight odour
- 4 Moderate odour
- 5 Strong odour

5 An acceptable odour is an odour rated at about 3 or less. Odour is evaluated on the basis of the almond odour that is characteristic of benzaldehyde. The presence of benzaldehyde is an indication of instability of the composition.

10 The following scale was used to evaluate clarity:

- 0 Completely clear (equivalent to the clarity of water)
- 1 Threshold translucence
- 2 Very slight translucence
- 15 3 Slight translucence
- 4 Moderate translucence
- 5 Opaque

A satisfactory clarity is a clarity rated at about 3 or less.

20 In addition, the compounds were observed to determine if they salted or became gritty during storage.

25 The composition of the invention can be used as an antiperspirant by applying an effective amount to decrease perspiration to the skin of a mammal that exhibits perspiration. The composition dries on the skin and has a pleasant feel.

30 The following non-limiting Examples are presented for purposes of illustration only and are not to be construed in a restrictive sense.

#### Example 1

20.00 Parts by weight of aluminium chlorohydrate was rapidly stirred into 25.00 parts by weight of propylene glycol in a stainless steel container equipped

with a Lightnin mixer and a hot water bath. Stirring was continued for 10 minutes and the solution was heated to 40°C. Stirring was continued until the solution became clear.

5           In a separate container, 19.30 parts by weight of propylene glycol, 1.50 parts by weight of potassium hydroxide, and 15.00 parts by weight of polyethylene glycol 200 were combined and heated to 125°C. 3.20  
10       Parts by weight of dibenzylidene sorbitol was feathered into the mixture with rapid  
stirring. Stirring was continued until the  
dibenzylidene sorbitol was dissolved.

          The aluminium chlorohydrate solution in the stainless steel container was heated to 95°C and added  
15       to the solution in the separate container.

          10.00 Parts by weight of deionized water and 5.00 parts by weight of a 40% aqueous solution of aluminium lactate with a pH of 5 were combined, heated to 95°C, and added to the solution in the separate container.  
20       1.00 Part by weight of fragrance was added at 95°C. The solution was packed at 90°C and allowed to cool.

          The resulting clear gel antiperspirant stick had a threshold almond odour rated at 1 on a scale of 0 to 5. The clarity of the resulting clear gel stick was rated  
25       as 0.5 on a scale of 0 to 5. Salting was observed on storage.

#### Example 2

          20.00 Parts by weight of aluminium chlorohydrate  
30       was rapidly stirred into 30.00 parts by weight of propylene glycol in a stainless steel container equipped with a Lightnin mixer and a hot water bath. Stirring was continued for 10 minutes and the solution was heated to 40°C. Stirring was continued until the solution  
35       became clear.

In a separate container, 23.40 parts by weight of propylene glycol, 1.50 parts by weight of 99% triethanolamine, and 6.00 parts by weight of methoxypolyethylene glycol 5000 were combined and heated to 125°C. 3.00 Parts by weight of dibenzylidene sorbitol was feathered into the mixture with rapid stirring. Stirring was continued until the dibenzylidene sorbitol was dissolved.

The aluminium chlorohydrate solution in the stainless steel container was heated to 95°C and added to the solution in the separate container.

13.00 Parts by weight of deionized water, 2.00 parts by weight of 0.5M potassium phosphate and 5.5% phosphoric acid in water, and 0.10 part by weight of 1% F,D & C Blue #1 in water were combined, heated to 95°C, and added to the solution in the separate container. 1.00 Part by weight of fragrance was added at 95°C. The solution was packed at 90°C and allowed to cool.

The resulting clear gel antiperspirant stick had a pH of 4.5 and was light blue-green in colour. It had a threshold almond odour rated at 1 on a scale of 0 to 5. The clarity of the resulting clear gel stick was rated as 1 on a scale of 0 to 5. Salting was observed on storage.

25

### Example 3

20.00 Parts by weight of aluminium chlorohydrate was rapidly stirred into 25.00 parts by weight of propylene glycol as described in Examples 1 and 2. In a separate container, 35.55 parts by weight of propylene glycol, 1.70 parts by weight of 50% potassium hydroxide, and 0.75 parts by weight of polyethylene glycol 5000 were combined and heated to 125°C. 2.00 Parts by weight of dibenzylidene sorbitol was feathered into the mixture with rapid stirring and dissolved as described. The

35

aluminium chlorohydrate solution in the stainless steel container was heated to 95°C and added to the solution in the separate container.

12.00 Parts by weight of deionized water, 0.90  
5 parts by weight of 1M citric acid and 2.00 parts by weight of 1M dipotassium phosphate were combined, heated to 95°C, and added to the solution in the separate container. 1.00 Part by weight of fragrance was added at 95°C. The solution was packed at 90°C and allowed to  
10 cool.

The resulting clear gel antiperspirant stick had a pH of 4.5. It had a barely noticeable almond odour rated at 0.5 on a scale of 0 to 5. The clarity of the resulting clear gel stick was rated as 3 on a scale of 0  
15 to 5. Salting was observed on storage.

#### Example 4

20.00 Parts by weight of aluminium chlorohydrate was rapidly stirred into 25.00 parts by weight of  
20 propylene glycol as described. In a separate container, 31.90 parts by weight of propylene glycol, 1.70 parts by weight of 50% potassium hydroxide, and 1.20 parts by weight of polyethylene glycol 5000 were combined and heated to 125°C. 2.70 Parts by weight of dibenzylidene  
25 sorbitol was feathered into the mixture and dissolved as described. The aluminium chlorohydrate solution in the stainless steel container was heated to 95°C and added to the solution in the separate container.

5.00 Parts by weight of deionized water, 10.00  
30 parts by weight of 0.5M potassium acid phthalate and 1.50 parts by weight of 50% potassium hydroxide were combined, heated to 95°C, and added to the solution in the separate container. 1.00 Part by weight of fragrance was added at 95°C. The solution was packed at  
35 90°C and allowed to cool.

The resulting clear gel antiperspirant stick had a pH of 4.5. It had a very slight almond odour rated at 2. The clarity of the resulting clear gel stick was rated as 2. Salting was observed on storage.

5

Example 5

20.00 Parts by weight of aluminium chlorohydrate was rapidly stirred into 25.00 parts by weight of propylene glycol as described. In a separate container, 10 33.70 parts by weight of propylene glycol, 1.50 parts by weight of triethanolamine, and 1.20 parts by weight of polyethylene glycol 5000 were combined and heated to 125°C. 2.50 Parts by weight of dibenzylidene sorbitol was feathered into the mixture and dissolved as 15 described. The aluminium chlorohydrate solution in the stainless steel container was heated to 95°C and added to the solution in the separate container.

13.80 Parts by weight of deionized water, 0.40 parts by weight of 1M citric acid, and 0.90 parts by 20 weight of 1M sodium citrate were combined, heated to 95°C, and added to the solution in the separate container. 1.00 Part by weight of fragrance was added at 95°C. The solution was packed at 90°C and allowed to cool.

25 The resulting clear gel antiperspirant stick had a pH of 4.2. It had a slight almond odour rated at 3. The clarity of the resulting clear gel stick was rated at 2. Salting was observed on storage.

30

Example 6

20.00 Parts by weight of aluminium chlorohydrate was rapidly stirred into 30.00 parts by weight of propylene glycol as described. In a separate container, 24.30 parts by weight of propylene glycol, 15.00 parts 35 by weight of glycerine and 1.00 part by weight of 99%

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